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Thermochemical study on ternary complex of dysprosium *m*-nitrobenzoic acid with *o*-phenanthroline

S. X. Xiao · J. J. Zhang · X. Li · L. J. Ye · H. W. Gu · N. Ren

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Abstract A ternary binuclear complex of dysprosium chloride hexahydrate with *m*-nitrobenzoic acid and 1,10-phenanthroline, $[Dy(m\text{-NBA})_3phen]_2{\cdot}4H_2O$ (*m*-NBA: *m*-nitrobenzoate; phen: 1,10-phenanthroline) was synthesized. The dissolution enthalpies of $[2phen{\cdot}H_2O(s)]$, [6*m*-HNBA(s)], $[2DyCl_3{\cdot}6H_2O(s)]$, and $[Dy(m\text{-NBA})_3phen]_2{\cdot}4H_2O(s)$ in the calorimetric solvent ($V_{DMSO}:V_{MeOH} = 3:2$) were determined by the solution–reaction isoperibol calorimeter at 298.15 K to be $\Delta_s H_m^\theta[2phen{\cdot}H_2O(s), 298.15\ K] = 21.7367 \pm 0.3150\ kJ{\cdot}mol^{-1}$, $\Delta_s H_m^\theta[6m\text{-HNBA}(s), 298.15\ K] = 15.3635 \pm 0.2235\ kJ{\cdot}mol^{-1}$, $\Delta_s H_m^\theta[2DyCl_3{\cdot}6H_2O(s), 298.15\ K] = -203.5331 \pm 0.2200\ kJ{\cdot}mol^{-1}$, and $\Delta_s H_m^\theta[[Dy(m\text{-NBA})_3phen]_2{\cdot}4H_2O(s), 298.15\ K] = 53.5965 \pm 0.2367\ kJ{\cdot}mol^{-1}$, respectively. The enthalpy change of the reaction was determined to be $\Delta_r H_m^\theta = 369.49 \pm 0.56\ kJ \cdot mol^{-1}$. According to the above results and the relevant data in the literature, through Hess' law, the standard molar enthalpy of formation of $[Dy(m\text{-NBA})_3phen]_2{\cdot}4H_2O(s)$ was estimated to be $\Delta_f H_m^\theta[[Dy(m\text{-NBA})_3phen]_2{\cdot}4H_2O(s), 298.15\ K] = -5525 \pm 6\ kJ{\cdot}mol^{-1}$.

Keywords Rare earth complex · Dysprosium chloride hexahydrate · *m*-Nitrobenzoic acid · 1,10-Phenanthroline · Thermochemical

Introduction

Since the late twentieth century drug-resistant bacteria have rapidly developed and spread all over the world, it has become one of the biggest health challenges the public health faced, while the development of bacteria-resistant drugs has lagged far behind the social needs. *m*-Nitrobenzoic acid ($C_7H_5O_4$, abbreviated to *m*-HNBA) is a common pharmaceutical intermediate; phenanthroline ($C_{12}H_8N_2$, abbreviated to phen), also called as 1,10-phenanthroline, which belongs to heterocyclic conjugated chelating ligands, can be used as anti-tumor drugs, DNA molecular probes, etc. after coordinated with metal ions [1]. The results of the deep research on biochemical effects and pharmacological effects of the rare earth complexes showed that rare earth have a strong affinity to many biological molecules and they could participate in some important life processes [2]; With an activation or inhibition effect for many enzymes and zymogens, rare earth also have the obvious sterilization, bacteriostasis and anti-tumor function [3–5]. Compared with lots of synthetic organic drugs and transition metal coordination compounds, the complexes of the rare earth with many organic compounds have a stronger sterilization and bacteriostasis effects, are of lower toxicity and lower accumulation in the body [6]. Therefore, rare earth anti-tumor antibiotics have a broad application prospect. In recent years, the research on synthesis and characterization of complex of rare earth coordinating with aromatic carboxylic acid and phenanthroline is increasingly active. Synthesis and thermal decomposition kinetics of the binuclear complex

S. X. Xiao · X. Li · L. J. Ye · H. W. Gu
Department of Chemistry and Life Science, Xiangnan University, Chenzhou 423000, People's Republic of China

J. J. Zhang (✉)
Experimental Center, Hebei Normal University, Shijiazhuang 050016, People's Republic of China
e-mail: jjzhang6@126.com

N. Ren
Department of Chemistry, Handan College, Handan 056005, People's Republic of China

have been reported in these articles [7–14], but the data of its standard molar enthalpy of formation still lacked. In this article, we synthesized a ternary binuclear complex of $[Dy(m\text{-}NBA)_3phen]_2 \cdot 4H_2O$ and the value of its standard molar enthalpy of formation was determined.

Experiment

Reagents and instruments

Dy_2O_3 (>99.9%, produced by the Chengdu Feitian Co., LTD); $DyCl_3 \cdot 6H_2O$ (synthesized by the reaction of Dy_2O_3 with 6.0 mol·L^{-1} HCl followed by means of water bath evaporation crystal); *m*-HNBA (>99.5%, A.R.); *o*-phenanthrolinemonohydrate (phen·H_2O, >99.5%, A.R., recrystallized with anhydrous ethanol); Dimethyl sulfoxide (DMSO, A.R.); methanol (MeOH, A.R.); ethanol (EtOH, A.R.); NaOH(A.R.); HCl (A.R.); KCl (calorimetric primary standard) of purity greater than 99.99% was dried in a vacuum oven for 6 h at 408.15 K.

Ultraviolet–visible spectrophotometer (U-3010, HITACHI, Japan); solution–reaction isoperibol calorimeter (SRC 100, constructed by the thermochemical laboratory of Wuhan University, China) [9]. The volume of the reaction vessel is 100 cm^3, the precision of the test temperature and control temperature are ±0.001 and ±0.0001 K, respectively.

Synthesis of complex $[Dy(m\text{-}NBA)_3phen]_2 \cdot 4H_2O(s)$

According to the reference [8], the synthesis and analysis of the complex can be summarized as follows.

$DyCl_3 \cdot 6H_2O$, *m*-HNBA and phen·H_2O were dissolved in 95% ethanol in a molar ratio of 1:3:1, respectively. The pH value of the *m*-HNBA was adjusted to 6–7 by adding 1.0 mol·L^{-1} NaOH solution. The ethanol solution of two ligands were mixed and then added dropwise to the ethanolic $DyCl_3 \cdot 6H_2O$ solution. At once a large white precipitate formed, the mixture solution was stirred for 8 h at room temperature and then deposited for 12 h. Subsequently, the precipitate was filtrated and washed several times with 95% ethanol. The product was dried under vacuum at 298.15 K until the weight of the crystals became a constant. Finally, the white powdery complex was obtained in 90.84% yield. The chemical composition of the synthetic sample was determined by elemental analysis for C, H, and N, by EDTA titration for Dy^{3+}, by TG-DTG analysis for H_2O. The analysis results proved that the composition of the complex was $[Dy(m\text{-}NBA)_3phen]_2 \cdot 4H_2O(s)$, and its purity was greater than 99.0%.

The solution–reaction isoperibol calorimeter and calibration

The solution–reaction isoperibol calorimeter with a constant temperature environment was constructed and calibrated according to the published literature [15]. The solution–reaction isoperibol calorimeter was calibrated by means of six para-experiments carried at 298.15 K using the solution of KCl dissolved in H_2O with a molar ratio of 1:1110 as standard substance. The actual dissolution enthalpies of six tests, $\Delta_s H_m^{\theta}[KCl(s),\ 298.15\ K] = (17597 \pm 17)\ J \cdot mol^{-1}$, which agreed with the published data $(17536 \pm 9)\ J \cdot mol^{-1}$ [16]. And the eventual error was less than 0.5%.

Determination of dissolution enthalpies

Thermochemical cycle of the synthetic reaction (1)

According to Hess's Law, the thermochemical cycle was designed as in Fig. 1.

The UV spectra and refractive indexes of the final solution of the reactants and the final solution of the products can be used to determine if they have the same thermodynamic state. In the present experiments, we determined the UV spectrum and refractive indexes of solution C and solution F in Fig. 1, and the experimental

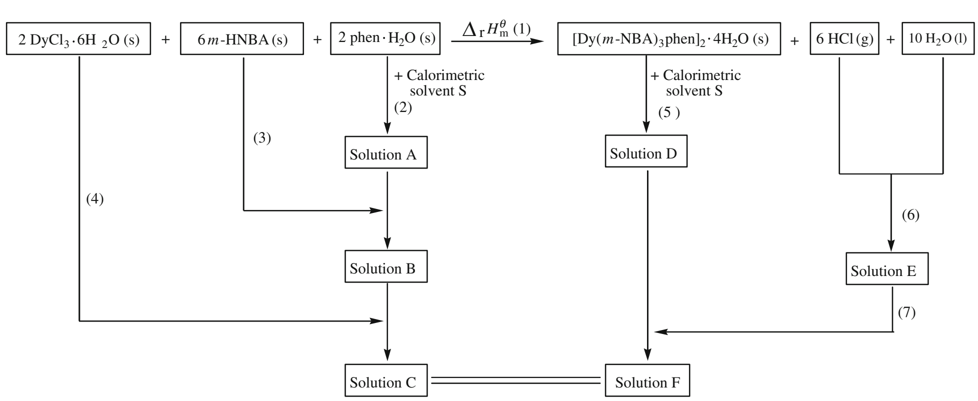

Fig. 1 Thermochemical cycle of the reaction (1)

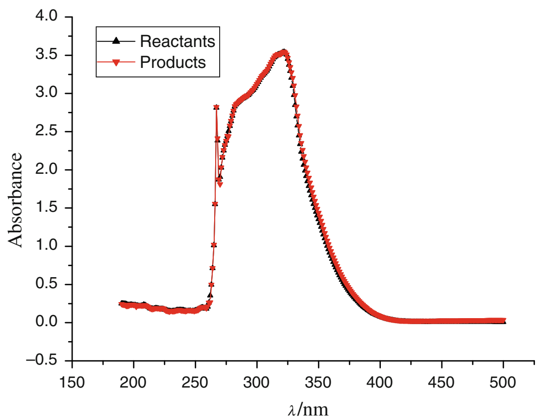

Fig. 2 UV spectrum of the final dissolution state of the reactants and products

results suggested that both of them have similar UV spectrum curves (Fig. 2) and equal refractive indexes ($\eta_{298.15\ K} = 1.4046$). It proves that they have the same thermodynamic state and that the thermochemical cycle of the reaction (1) designed is reliable.

The choice of solvent

It is very important to choose the calorimetric solvent which must dissolve the chemicals in the sample cell completely and very rapidly. The mixture solvent (S) of DMSO and MeOH (V_{DMSO}:V_{MeOH} = 3:2) is the most appropriate solvent for this experiment.

Determination of dissolution enthalpies of all the chemicals in synthetic reaction (1)

Both the calorimeter's calibration and enthalpy of dissolution of the sample were determined under the same conditions. The experimental temperature was 298.15 K, current was 11.7600 mA, and the resistance of the heater was 1251.6 Ω.

The phen·H_2O(s) was grinded in an agate mortar, and a sample (0.0496 g) was placed into a sample cell in calorimeter. The calorimetric solvent S (100 cm^3) was added into the reaction vessel. Calorimeter was adjusted to a constant temperature of 298.15 K, and the dissolution enthalpy of reaction (2) was determined by a series of five para-experiments.

$$2\ \text{phen}\cdot H_2O(s) + \text{solvent S} \rightarrow \text{solution A.} \quad (2)$$

The solution A above was kept in the reaction vessel, and powdery *m*-HNBA(s) (0.1253 g) was put into the sample cell. Then the calorimeter was adjusted to a constant temperature of 298.15 K and the dissolution enthalpy of reaction (3) was determined by a series of five para-experiments.

$$6\ m\text{-HNBA(s)} + \text{solution A} \rightarrow \text{solution B.} \quad (3)$$

The solution B above was kept in the reaction vessel, and powdery $DyCl_3 \cdot 6H_2O$(s) (0.0942 g) was put into the sample cell. Then the calorimeter was adjusted to a constant temperature of 298.15 K, and the dissolution enthalpy of reaction (4) was determined by a series of five para-experiments.

$$6\ DyCl_3 \cdot 6H_2O(s) + \text{solution B} \rightarrow \text{solution C.} \quad (4)$$

Powdered [Dy(*m*-NBA)$_3$phen]$_2$·4H_2O(s) (0.2193 g) was put into the sample cell in calorimeter, and the calorimeter solvent S (100 cm^3) was added into the reaction vessel. Calorimeter was adjusted to a constant temperature of 298.15 K, and the dissolution enthalpy of reaction (5) was determined by a series of five para-experiments.

$$[Dy(m\text{-NBA})_3\text{phen}]_2\cdot 4H_2O(s) + \text{solvent S} \rightarrow \text{solution D.} \quad (5)$$

A mass of 1.5 mmol HCl(g) was dissolved into 2.5 mmol H_2O(l).

$$6\ HCl(g) + 10\ H_2O(l) \rightarrow \text{solution E.} \quad (6)$$

The solution D above was kept in the reaction vessel. The solution E was put into the sample cell. Then the calorimeter was adjusted to a constant temperature of 298.15 K, and the dissolution enthalpy of reaction (7) was determined by a series of five para-experiments.

$$\text{solution D} + \text{solution E} \rightarrow \text{solution F.} \quad (7)$$

The calorimetric results of reactions (2–5) and (7) are listed in Table 1.

Results and discussion

Results of the calorimetric experiment

The dissolution enthalpies of 2 phen·H_2O(s), 6*m*-HNBA(s), 2 $DyCl_3 \cdot 6H_2O$(s), and [Dy(*m*-NBA)$_3$phen]$_2$·4H_2O(s) in the calorimetric solvent S are given in Table 1 (298.15 K, R = 1251.6 Ω, I = 11.7600 mA).

Evaluation of $\Delta_d H_m^\theta(6)$

According to reaction (6)

$$6\ HCl(g) + 10\ H_2O(l) \rightarrow \text{solution E.} \quad (6)$$

The molality of solution E is 33.33 mol·kg^{-1}. According to the relationship between the enthalpy of dilution and the dissolution enthalpy

$$\Delta_d H_m^\theta = \Delta_s H_m^\theta(\text{diluted}) - \Delta_s H_m^\theta\ (\text{concentrated}).$$

Table 1 Dissolution enthalpies of [2 phen·H_2O(s)], [6*m*-HNBA(s)], [2 $DyCl_3 \cdot 6H_2O$(s)] and [Dy(*m*-NBA)$_3$ phen]$_2$·$4H_2O$(s) in the calorimetric solvent S at 298.15 K

Systems	No.	m/g	t/s	$\Delta_s H_m^\theta/\text{kJ}\cdot\text{mol}^{-1}$	Avg.$\Delta_s H_m^\theta/\text{kJ}\cdot\text{mol}^{-1}$
2 phen·H_2O(s) in solvent S	1	0.0495	20.11	21.3786	21.7367 ± 0.3150
	2	0.0494	29.81	21.4203	
	3	0.0499	19.80	21.9205	
	4	0.0501	30.13	22.0667	
	5	0.0492	20.33	21.8973	
6*m*-HNBA(s) in solution A	1	0.1257	24.86	15.1560	15.3635 ± 0.2235
	2	0.1255	20.13	15.5081	
	3	0.1249	27.48	15.3299	
	4	0.1248	25.02	15.6668	
	5	0.1252	20.19	15.1566	
$2DyCl_3 \cdot 6H_2O$(s) in solution B	1	0.0945	174.93	−203.5124	−203.5331 ± 0.2200
	2	0.0952	171.08	−203.4946	
	3	0.0941	184.84	−203.2074	
	4	0.0948	160.33	−203.6499	
	5	0.0957	173.37	−203.8014	
[Dy(*m*-NBA)$_3$ phen]$_2$·$4H_2O$(s) in solvent S	1	0.2197	34.49	53.7956	53.5965 ± 0.2367
	2	0.2197	35.10	53.4022	
	3	0.2193	33.10	53.3544	
	4	0.2198	37.30	53.5419	
	5	0.2195	36.47	53.8884	
Solution D + solution E	1	0.0259	217.09	−317.3866	−317.5467 ± 0.2321
	2	0.0290	208.08	−317.6632	
	3	0.0259	214.81	−317.8988	
	4	0.0288	201.49	−317.4408	
	5	0.0290	199.65	−317.3439	

So that

$$\Delta_d H_m^\theta = \Delta_s H_m^\theta\left(m = 1.00\ \text{mol}\cdot\text{kg}^{-1}\right) - \Delta_s H_m^\theta\left(m = 33.33\ \text{mol}\cdot\text{kg}^{-1}\right).$$

According to the data in reference [17], the enthalpy of extremely diluted HCl(g) is

$$\Delta_s H_m^\theta(\text{HCl(g)},\ 298.15\ \text{K}) = -74.84\ \text{kJ}\cdot\text{mol}^{-1}.$$

According to the relationship between the apparent molar enthalpy and integration enthalpy of extremely diluted solution: $\Delta_d H^\theta_{c\to 0} = -\Phi_{L2}$.

According to the data in reference [18]

$$\Delta_d H^\theta_{(1.00\to 0)} = -\Phi_{L(1.00\to 0)} = -1.70\ \text{kJ}\cdot\text{mol}^{-1},$$

$$\Delta_d H^\theta_{(33.33\to 0)} = -\Phi_{L(33.33\to 0)} = -31.21\ \text{kJ}\cdot\text{mol}^{-1}.$$

So that

$$\begin{aligned}\Delta_d H^\theta_{(33.33\to 1.00)} &= \Delta_d H^\theta_{(33.33\to 0)} - \Delta_d H^\theta_{(1.00\to 0)} \\ &= -\Phi_{L(33.33\to 0)} + \Phi_{L(1.00\to 0)} \\ &= -31.21 + 1.70 = -29.51\ \text{kJ}\cdot\text{mol}^{-1}.\end{aligned}$$

So that

$$\begin{aligned}\Delta_s H_m^\theta(6) &= \Delta_s H_m^\theta(m = 33.33\ \text{mol}\cdot\text{kg}^{-1}) \\ &= \Delta_s H_m^\theta(m = 1.00\ \text{mol}\cdot\text{kg}^{-1}) - \Delta_s H^\theta_{m(33.33\to 1.00)} \\ &= [-74.84 - (-29.51)]\text{kJ}\cdot\text{mol}^{-1} \\ &= -45.33\ \text{kJ}\cdot\text{mol}^{-1}.\end{aligned}$$

The standard molar enthalpy change of reaction (1)

According to Hess's law, the standard molar reaction enthalpy of the reaction is obtained.

$$\begin{aligned}\Delta_r H_m^\theta(1) &= \Delta_s H_m^\theta(2) + \Delta_s H_m^\theta(3) + \Delta_s H_m^\theta(4) - \Delta_s H_m^\theta(5) \\ &\quad - \Delta_s H_m^\theta(6) - \Delta_s H_m^\theta(7) \\ &= [21.7367 + 15.3635 - 203.5331 \\ &\quad - 53.5965 + 45.33 + 317.5467] \\ &\quad \pm \left[\sqrt{0.3150^2 + 0.2235^2 + 0.2200^2 + 0.2367^2 + 0.2321^2}\right] \\ &= 369.49\ \pm\ 0.56\ \text{kJ}\cdot\text{mol}^{-1}.\end{aligned}$$

Estimation of $\Delta_f H_m^\theta$[[Dy(*m*-NBA)$_3$phen]$_2$·4H$_2$O(s), 298.15 K]

According to Hess's law

$$\begin{aligned}\Delta_r H_m^\theta(1) =& \Delta_f H_m^\theta\left[\left[\mathrm{Dy}(m\text{-NBA})_3\mathrm{phen}\right]_2\cdot 4\mathrm{H_2O(s)}, 298.15\ \mathrm{K}\right] \\ &+ 6\Delta_f H_m^\theta[\mathrm{HCl(g)},\ 298.15\ \mathrm{K}] + 10\Delta_f H_m^\theta[\mathrm{H_2O(l)},\ 298.15\ \mathrm{K}] \\ &- 2\Delta_f H_m^\theta[\mathrm{DyCl_3}\cdot 6\mathrm{H_2O(s)},\ 298.15\ \mathrm{K}] \\ &- 6\Delta_f H_m^\theta[m\text{-}\mathrm{HNBA(s)},\ 298.15\ \mathrm{K}] \\ &- 2\Delta_f H_m^\theta[\mathrm{phen}\cdot\mathrm{H_2O(s)},\ 298.15\ \mathrm{K}].\end{aligned}$$

According to reference [18–21]

$$\Delta_f H_m^\theta[\mathrm{HCl(g)}, 298.15\,\mathrm{K}] = -92.31 \pm 0.10\,\mathrm{kJ\cdot mol^{-1}}$$
$$\Delta_f H_m^\theta[\mathrm{H_2O(l)}, 298.15\,\mathrm{K}] = -285.83 \pm 0.04\,\mathrm{kJ\cdot mol^{-1}}$$
$$\Delta_f H_m^\theta[\mathrm{DyCl_3\cdot 6H_2O(s)}, 298.15\,\mathrm{K}] = -2870\,\mathrm{kJ\cdot mol^{-1}}$$
$$\Delta_f H_m^\theta[m\text{-HNBA(s)}, 298.15\,\mathrm{K}] = -414.0 \pm 0.4\,\mathrm{kJ\cdot mol^{-1}}$$
$$\Delta_f H_m^\theta[\mathrm{phen\cdot H_2O(s)}, 298.15K] = -391.34 \pm 2.98\,\mathrm{kJ\cdot mol^{-1}}.$$

And the above-calculated value of

$\Delta_r H_m^\theta(1) = 369.49 \pm 0.56\ \mathrm{kJ\cdot mol^{-1}}$.

So that

$$\begin{aligned}&\Delta_f H_m^\theta\left[\left[\mathrm{Dy}(m\text{-NBA})_3\mathrm{phen}\right]_2\cdot 4\mathrm{H_2O(s)},\ 298.15\ \mathrm{K}\right] \\ &= [369.49 - 6\times(-92.31) - 10\times(-285.83) \\ &\quad + 2\times(-2870) + 6\times(-414.0) + 2\times(-391.34)] \\ &\quad \pm\left[\sqrt{0.56^2 + (6\times 0.10)^2 + (10\times 0.04)^2 + (6\times 0.4)^2 + (2\times 2.98)^2}\right] \\ &= -5225 \pm 6\ \mathrm{kJ\cdot mol^{-1}}.\end{aligned}$$

Conclusions

In this article, a ternary solid complex [Dy(*m*-NBA)$_3$phen]$_2$·4H$_2$O was synthesized, and the study on its thermodynamic properties was carried out. The dissolution enthalpies of relevant substances were determined, respectively, by the solution–reaction isoperibol calorimeter at 298.15 K. The calculated results based on experimental data indicated the standard molar enthalpy of reaction of the synthesis of the complex was determined to be $\Delta_r H_m^\theta = 369.49 \pm 0.56\ \mathrm{kJ\cdot mol^{-1}}$ and the standard molar enthalpy of formation of [Dy(*m*-NBA)$_3$phen]$_2$·4H$_2$O was estimated to be $\Delta_f H_m^\theta$[[Dy(*m*-NBA)$_3$phen]$_2$·4H$_2$O(s), 298.15 K] = −5525 ± 6 kJ·mol^{-1}.

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